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Effects of quaternary ammonium-methacrylates on the mechanical properties of unfilled resins

Tomohiro Hoshika^a, Yoshihiro Nishitani^a, Masahiro Yoshiyama^a,
William O. Key III^b, William Brantley^b, Kelli A. Agee^b, Lorenzo Breschi^c,
Milena Cadenaro^d, Franklin R. Tay^e, Frederick Rueggeberg^b,
David H. Pashley^{b,*}

^a Department of Operative Dentistry, Okayama University Graduate School of Medicine, Dentistry and Pharmaceutical Sciences, Okayama, Japan

^b Department of Oral Biology, Georgia Regents University, College of Dental Medicine, Augusta, GA, USA

^c Department of Biomedicine, University of Trieste and IGM-CNR, Unit of Bologna, c/o IOR, Bologna, Italy

^d Department of Biomedicine, Unit of Dental Sciences and Biomaterials, University of Trieste, Trieste, Italy

^e Department of Endodontics, Georgia Regents University, College of Dental Medicine, Augusta, GA, USA

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ABSTRACT

Objective. Adding antimicrobial/anti-MMP quaternary ammonium methacrylates (QAMs) to comonomer blends should not weaken the mechanical properties of dental resins. This work evaluated the degree conversion and mechanical properties of BisGMA/TEGDMA/HEMA (60:30:10) containing 0–15 mass% QAMs A–E (A: 2-acryloxyethyltrimethyl ammonium chloride; B: [3-(methacryloylamino)propyl]trimethylammonium chloride; C: [2-(methacryloxy)ethyl] trimethyl ammonium chloride; D: diallyldimethyl ammonium chloride; E: 2-(methacryloyloxy) ethyltrimethyl ammonium methyl sulfate.

Methods. Unfilled resins with and without QAM were placed on ATR-FTIR and light-polymerized for 20 s in a thin film at 30 °C. Unfilled resin beams were casted from square hollow glass tubings. Half of the beams were tested after 3 days of drying (control); the other half were tested wet after 3 days of water storage.

Results. Addition of QAMs in control resins significantly increased conversion 600 s after light termination, with the exception of 5% MAPTAC ($p < 0.05$). Increase of QAM content within a formulation significantly increased conversion. Control beams gave dry Young's moduli of ~700 MPa. Addition of 5, 10 or 15 mass% QAMs produced significant reductions in dry Young's moduli except for 5% B or C. 15 mass% A, B and C lowered the wet Young's moduli of the resin beams by more than 30%. The ultimate tensile stress (UTS) of control dry resin was 89 ± 11 MPa. Addition of 5–10 mass% QAMs had no adverse effect on the dry UTS. After water storage, the UTS of all resin blends fell significantly ($p < 0.05$), especially when 15 wt% QAMs was added. Control dry beams gave fracture toughness (K_{IC}) values of 0.88 ± 0.1 MPa m^{1/2}. Wet values were significantly higher at 1.02 ± 0.06 ($p < 0.05$). K_{IC} of dry beams varied from 0.85 ± 0.08 at 5% QAMs to 0.49 ± 0.05 at 15% QAMs. Wet beams gave K_{IC} values of 1.02 ± 0.06 MPa m^{1/2} that fell to 0.23 ± 0.01 at 15% QAMs.

* Corresponding author at: Department of Oral Biology, College of Dental Medicine, Georgia Health Sciences University, Augusta, GA 30912-1129, USA. Tel.: +1 706 721 2031; fax: +1 706 721 6252.

E-mail address: dpashley@gru.edu (D.H. Pashley).

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Significance. Addition of 10% QAMs increased the degree of conversion of unfilled resins, but lowered wet toughness and UTS; addition of 15% QAMs lowered the mechanical properties of wet resins below acceptable levels.

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1. Introduction

Quaternary ammonium methacrylates (QAMs) have been added to dental comonomer blends for their antibacterial [1–5] and anti-MMP activity [6,7]. Because most QAMs are monomethacrylates, it is important to determine if addition of QAMs to comonomer blends cause any weakening of their mechanical properties, and if so, at what concentration. A good example of a QAM used in a dental product is 12-methacryloyloxydodecylpyridinium bromide (MDPB). It is used in Clearfil SE Protect as an antibacterial primer [1–3,8].

It is well-known that water sorption into hydrophilic polymers plasticizes them and lowers their mechanical properties [9–13]. Unfilled resins tend to have low mechanical properties values because they have no reinforcing fillers. The lower flexural strength and flexural modulus of unfilled resins results in a lower fracture toughness [14], giving them values similar to compomers [14,15].

As one incorporates any monomethacrylate into comonomer blends at the expense of dimethacrylates, the degree of cross-linking of the polymer decreases. This results in lower flexural strength and flexural modulus. Presumably, this should also lower other mechanical properties of resins.

The purpose of this study was to determine if addition of 5, 10 or 15 wt% QAMs to a BisGMA/TEGDMA/HEMA blend would lower the degree of conversion or mechanical properties of beam specimens of unfilled resins. The first null test hypothesis was that (1) addition of 5, 10 or 15 wt% QAMs has no effect on the degree of conversion or dimethacrylate blends, (2) addition of 5, 10 or 15 wt% QAMs has no effect on the mechanical properties of dimethacrylate blends, (3) that addition of 5, 10 or 15% QAMs to dimethacrylate blends immersed in water does not produce water-sorption induced changes in the mechanical properties of QAM-containing dimethacrylates.

2. Materials and methods

2.1. Degree of conversion

Before testing the effect of QAM-containing adhesives on the mechanical properties of unfilled resins, a careful investigation of the effect of incorporation of QAMs into dental adhesives is necessary to verify their role in polymerization. Indeed, it was clearly shown that the degree of conversion (DC) of dental adhesives is an important parameter since low mechanical properties associated with monomer elution are related with low percentage of monomer to polymer conversion within resin-based materials [16–18].

Previous investigations showed that DC depends on resin type, hydrophilicity, solvent content and photo-initiator blended within the monomers. It has been shown that even though camphoroquinone (CQ) is the most common photo-initiating system in dental resin restorative materials [19], it may impair the polymerization of hydrophilic monomers due to CQ's intrinsic hydrophobic nature. On the other hand, the use of CQ-alternative photoinitiators, such as diphenyl(2,4,6-trimethylbenzoyl)-phosphine oxide, TPO, may improve the extent of polymerization (E_p) of hydrophilic adhesive formulations [20].

One drop of each resin mixture was placed between two, 0.15-mm thick, laterally placed microscope cover glass plates acting as shims, directly on the surface of a diamond crystal in a horizontal attenuated total reflectance heated unit (25 °C) stage (Golden Gate Mk II, SPECAC Inc., Cranston, RI) using a 1 mL disposable syringe (Norm-Ject, Tuttlingen, Germany). The attachment was positioned in the optical compartment of a Fourier transform infrared spectrophotometer (FTS-40, Digilab/BioRad, Cambridge, MA). A 1.5 cm × 1.5 cm × 76 μm piece of polyester film (Mylar, Type D, Polymer Plastics Corporation, Reno, NY, USA) was immediately placed over the top of the deposited resin (covering the two glass shims laterally) to exclude oxygen (Fig. 1), and a 3-mm thick, clear glass plate was positioned over the top of the assembly and pressed down, forcing the fluid to spread and attain the final thickness of the two lateral glass shims. The flat end of the light guide of a quartz-tungsten-halogen light-curing unit (Optilux 401, Demetron/Kerr, Danbury, CT) was placed coincident with the underlying diamond element, and directly against the upper glass surface. The irradiance of the curing light was measuring through the same 3-mm thick glass plate, using a laboratory grade spectroradiometric system, consisting of a 6" NIST-traceable integrating sphere (Labsphere, North Sutton, NH) and a spectrometer (USA2000, Ocean Optics, Dunedin, FL)

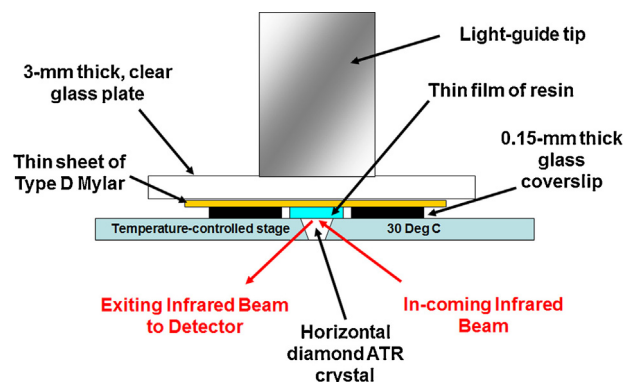


Fig. 1 – Schematic diagram of test device for obtaining infrared spectra.

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