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High-temperature high-pressure polymerized urethane dimethacrylate—Mechanical properties and monomer release



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ARTICLE INFO

Article history: Received 26 September 2013 Received in revised form 6 December 2013 Accepted 17 December 2013

Keywords: High-temperature high-pressure polymerization UDMA Mechanical properties Physical properties Monomer release

ABSTRACT

Objective. This study was conducted to determine selected mechanical/physical properties of and monomer release from high-temperature high-pressure (HT/HP) polymerized urethane dimethacrylate (UDMA).

Methods. Flexural strength (σ_f), hardness, fracture toughness (K_{IC}), and density (ρ) were determined for five UDMA resin blocks produced via different polymerization protocols. High performance liquid chromatography (HPLC) was used to determine monomer release from the five polymers. One way ANOVA, Scheffé multiple means comparisons ($\alpha = 0.05$), and Weibull statistics (for σ_f) were used to analyze the results.

Results. The results showed that HT/HP polymerization resulted in a significant (p < 0.05) increase in σ_f and ρ , along with an increase in Weibull modulus. No significant differences were found in hardness and K_{IC} between the two HT/HP polymerized materials. A significantly lower (p < 0.05) monomer release was detected for the HT/HP polymerized groups.

Significance. The results of this study suggest that HT/HP polymerization affects the network structure and leads to UDMA polymers with improved mechanical/physical properties and with dramatically reduced monomer release. The low elution of monomers from HT/HP and HP polymerized materials suggests the achievement of a higher degree of conversion and a lesser degree of inhomogeneity with regards to microgel domains. The results, however, cannot fully explain the dramatic increase in mechanical/physical properties reported previously for RCB, improvements that may be due to a better filler-matrix interaction afforded by HT/HP polymerization.

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1. Introduction

It took 40 years for computer-aided design/computer aided manufacturing (CAD/CAM) to become a widely spread

technology in dentistry [1]. From the dental laboratory perspective, it offers several advantages, including automation of fabrication procedures, increased quality, minimized inaccuracies, and faster delivery [2]. Ceramic and resin composite

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^{0109-5641/\$ –} see front matter © 2013 Academy of Dental Materials. Published by Elsevier Ltd. All rights reserved. http://dx.doi.org/10.1016/j.dental.2013.12.009

blocks for CAD/CAM-produced indirect dental restorations is a fast growing segment of the dental material market [3,4].

While ceramic blocks, due to their superior aesthetics and mechanical properties have been the most used materials for CAD/CAM, easier machinability, considerable reduction in manufacturing time and wear on cutting tools, purportedly easier repair, and lower cost lead to the development of resin composite blocks (RCB) as a viable alternative [3,5]. The three main constituents of dental resin composites are the matrix, the filler, and a coupling agent that provides bonding between them. Commonly used monomers in the resin matrix are bisphenol A-glycidyl methacrylate (Bis-GMA) and urethane dimethacrylate (UDMA), combined with triethylene glycol dimethacrylate (TEGDMA) to adjust viscosity. As part of the continuous effort to improve the mechanical properties of RCB, we have recently reported on the dramatic effect of high-temperature high-pressure (HT/HP) polymerization on the properties of commercial [6] and experimental resin composites [7].

The aim of this study was to assess the effect of HT/HP polymerization on the mechanical properties of and monomer release from the polymer alone, in order to better understand the results obtained for resin composites. Due to concerns related to the toxicity of bis-phenol A (BPA), we decided to base our experimental composites on UDMA [7]. Other advantages of UDMA over Bis-GMA are its lower molecular weight (and consequently higher concentration of double bonds) as well as lower viscosity due to the presence of flexible urethane or carbamate linkages and absence of aromatic groups, which may improve toughness of the polymer/composite [8,9]. The null hypotheses tested were: (1) mechanical/physical properties of and monomer release from HT/HP polymerized UDMA are not different from those of control thermo-cured or lightcured UDMA polymers; (2) mechanical/physical properties of and monomer release from HP/HT polymerized UDMA are not affected by the presence/absence of an initiator.

2. Materials and methods

The monomer used in this study was UDMA (MW = 470.56; CAS 72869-86-4; Esstech, Germany). Table 1 summarizes the materials, the polymerization parameters, and the group designation of the five UDMA polymers made, while the experimental details are given below.

2.1. Control, light-cured UDMA (group LC)

The monomer was mixed with 100 ppm hydroquinone monomethyl ether (HQME, Fluka, France), 0.5% (weight)

camphoroquinone (CQ, Aldrich, Germany), and 1% (weight) 4,N,N-trimethylanilin (TMA, Aldrich, Germany) in a planetary mixer (Thinky AR 250, Tokyo, Japan). The mix was left at room temperature for 1 day before being cast into $(2 \times 4 \times 20)$ mm silicone molds. Light-curing was performed three times (ones in the middle of the sample and once on each extremity of the sample) for 40 s each, with a LED curing unit (Radii, SDI, Victoria, Australia) operated at a power density of 1200 mW/cm² (measured with a curing radiometer, Dentsply Caulk, Milford, USA).

2.2. Control, thermo-cured UDMA (group TC)

The monomer was mixed with initiator, 0.5% benzoyl peroxide (BPO; Sigma Aldrich, Steinheim, Germany); 100 g was placed inside a flexible silicone tube (25 mm internal diameter) and was thermally cured at 90 °C, at ambient pressure (0.1 MPa), in a furnace (Memmert, Schwabach, Germany), for 4 h.

2.3. Experimental, HP cured UDMA (group 90I300)

The composition (monomer with 0.5% BPO) and curing temperature (90 °C) for this group was the same as that of the **TC** control group, the difference being the high pressure during curing. The monomer-initiator mix (100 g) was placed inside a flexible silicone tube (25 mm internal diameter) and cured at 90 °C under high pressure (300 MPa), in a custom-built autoclave, for 4 h.

2.4. Experimental, HT/HP cured UDMA (group 190I300)

The composition (monomer with 0.5% BPO) for this group was the same as that of the **TC** control group, the difference being the high temperature and high pressure during curing. This group differed from **90I300** group by the high temperature during curing. The monomer-initiator mix (100 g) was placed inside a flexible silicone tube (25 mm internal diameter) and cured at 190 °C under high pressure (300 MPa), in a custom-built autoclave, for 1 h.

2.5. Experimental, no initiator HT/HP cured UDMA (group 190NI300)

This group differed from the experimental **190I300** group by the absence of an initiator. The monomer (100 g) was placed inside a flexible silicone tube (25 mm internal diameter) and

Table 1 – Experimental group, materials, and polymerization parameters.				
Group	Manufacturer	Polymer*	Initiator#	Polymerization parameters
LC	Laboratory	100% UDMA	0.5% CQ+ 1% 4,N,N-TMA + 100 ppm MEHQ	Light-cured
TC	Laboratory	100% UDMA	0.5% PBO	90 °C, 0.1 MPa for 4 h
90I300	Laboratory	100% UDMA	0.5% PBO	90 °C, 300 MPa for 4 h
190I300	Laboratory	100% UDMA	0.5% PBO	190 °C, 300 MPa for 1 h
190NI300	Laboratory	100% UDMA	None	190 $^\circ\text{C},$ 300 MPa for 1 h

* UDMA is urethane dimethacrylate.

[#] BPO is benzoyl peroxide; CQ is camphoroquinone; TMA is 4,N,N-trimethylanilin; HQME is hydroquinone monomethyl ether.

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