



Monomer elution in relation to degree of conversion for different types of composite



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ABSTRACT

Objectives: The aim of this study was to determine the degree of conversion (DC) and the monomer release of three composite types when employed following a layer- and bulk-filling technique.

Methods: The release of monomers from a 'conventional paste-like' (Filtek Z250), a 'conventional flowable' (Filtek Supreme XTE Flowable) and a 'bulk-fill' flowable composite (Filtek Bulk Fill Flowable) from the same manufacturer (3M ESPE, Seefeld, Germany) was determined. Ten cylindrical specimens per composite were built, either in two 2-mm layers or in one 4-mm bulk. DC was measured at the specimen top and bottom surface using micro-Raman spectroscopy, after which the specimens were immersed in 2 ml absolute ethanol for 24 h at 37 °C. This solution was refreshed weekly during six weeks and the concentration of BisGMA, BisEMA(6), BisPMA, UDMA, TEGDMA and BPA was determined by liquid chromatography/mass spectroscopy.

Results: DC at the specimen top and bottom was similar except for the bulk-fill technique, which resulted in significantly lower DC at the specimen bottom. The release of BisGMA and TEGDMA was initially very high, but rapidly dropped in the second week. In contrast, the release of BisPMA and UDMA increased initially, but then declined towards the sixth week. BisEMA(6) release was relatively steady over time. All composites released small amounts of BPA. The total monomer release was significantly lower for the layer- than the bulk-filling technique.

Conclusions: The slightly reduced degree of conversion at 4-mm depth resulted in a higher monomer elution when the composite was applied following a bulk-fill application method.

Clinical significance: Applying a flowable and a bulk-fill composite following a bulk-fill application method resulted in a significantly reduced degree of conversion at the bottom of polymerized composite specimens when compared to a layer-application method. This reduced polymerization degree was reflected in significantly increased monomer release.

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1. Introduction

In order to polymerize sufficiently and to reduce polymerization shrinkage stress, it has been recommended to apply dental composite incrementally, commonly in 2-mm thick layers. The

more recently introduced 'bulk-fill' composites can be applied and cured properly in 4-mm thick layers. Filling cavities with composite in bulk became feasible thanks to reduced shrinkage-stress during polymerization and deeper curing. Typically, bulk-fill composites contain new monomers [1], more translucent filler [2] and new photo-initiator systems [3].

As bulk-fill composites are intended to fill deep cavities with the cavity bottom close to the pulp, some biocompatibility concerns may arise in particular when the composite would not cure properly at the cavity bottom, so that monomers can leach out in

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Table 1

List of the three composites investigated and their composition.

Composite	Shade	Composition ^{a,b}	Filler size ^a	Lot number
Filtek Z250 Universal	A3	1–10 wt% BisGMA, <5 wt% TEGDMA, 1–10 wt% UDMA, 1–10 wt% BisEMA(6), 75–85 wt% silane treated ceramic filler	0.01–3.5 μm zirconia/silica filler (average 0.6 μm)	N446661
Filtek Supreme XTE Flowable	A3	5–10 wt% BisGMA, 5–10 wt% TEGDMA, 15–25 wt% BisPMA, 50–60 wt% silane treated ceramic filler, 5–10 wt% silane treated silica filler	0.02 μm non-agglomerated/ non-aggregated silica filler, 0.005–0.01 μm zirconia filler	N445013
Filtek Bulk Fill Flowable	A3	1–10 wt% BisGMA, <1 wt% TEGDMA, 10–20 wt% UDMA, 1–10 wt% BisEMA(6), 10–20 wt% BisPMA, 50–60 wt% silane treated ceramic filler, 5–10 wt% silane treated silica filler	0.01–3.5 μm zirconia/silica filler (average 0.6 μm), 0.1–5.0 μm ytterbium trifluoride	N426393

^a According to technical information provided by the manufacturer 3M ESPE.^b Abbreviations: BisGMA, bisphenol A-glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; BisEMA(6), bisphenol A ethoxylateddimethacrylate; BisPMA, 2,2-Bis-(4-(3 methacryloxypropoxy) phenyl) propane.

the oral cavity and easily reach the pulpal tissues. Methacrylate monomers are very reactive in nature, and in particular *in-vitro* research has shown that they may adversely interact with oral cells. It has been revealed that they may disturb the redox homeostasis through the generation of reactive oxygen species, thereby seriously disturbing vital cell functions [4]. They are not only cytotoxic at high concentrations [5,6], but they have also been associated with genotoxicity [7,8]. In this regard, the polymerization efficiency is important, as composites with low polymerization degree release more monomers [9].

Polymerization efficiency is best measured chemically in terms of the degree of monomer conversion (DC) using Fourier Transform infrared spectroscopy (FTIR) or micro-Raman spectroscopy (μRaman). In particular, the ratio of DC measured at the bottom to that at the top surface was found to correlate well with surface micro-hardness, typically measured as an indirect evaluation of the polymerization efficiency [10]. Curing depth is affected by many factors such as, among others, the irradiated light-output, the uniformity of the light bundle, the distance to the composite surface, the light attenuation by the composite and the surrounding tissue, and the light transmittance through the composite [11–13].

Apart from monomers, composites may also release other compounds, the most controversial being Bisphenol A (BPA) [14–17]. Since BPA is used to synthesize some very frequently used monomers in composites, such as BisGMA and BisEMA, composites may unintentionally contain BPA as a residue of the monomer synthesis process. Being a mild xeno-oestrogen, BPA has mainly been associated with reproductive toxicity [18].

The objective of this study was to determine DC and the release of monomers of three different types of composite when employed following a layer- and bulk-filling technique, and to determine whether any relationship between DC and the release of monomers may exist up to six weeks. The null hypotheses tested

were (1) that no difference in DC and in the overall release of monomers was measured for the three different composite types and (2) that the application technique employed (bulk filling versus layer filling) did not influence the degree of conversion and monomer release.

2. Materials and methods

A conventional restorative composite with a paste-like consistency (Filtek Z250 Universal), a 'conventional flowable' composite (Filtek Supreme XTE Flowable) and a 'bulk-fill flowable' composite (Filtek Bulk Fill Flowable), all from the same manufacturer (3M ESPE, Seefeld, Germany), were selected (Table 1). The monomer composition of the three composites is alike and could be retrieved from the technical information provided by the manufacturer (MSDS).

Cylindrical samples were prepared of each composite type ($n = 10$) using a teflon mold (5-mm diameter, 4-mm depth) that was filled with composite in two 2-mm thick layers or in one 4-mm bulk. The top and the bottom were covered with a glass slide to prevent oxygen inhibition. Each of the two layers and the bulk were cured for 20 s using a polywave LED light-curing unit (Bluephase 20i, Vivadent Ivoclar, Schaan, Liechtenstein). Irradiance at the tip was measured using the MARC[®] Patient Simulator (BlueLight Analytics, Halifax, NS, Canada) to be around 1.100 mW/cm².

After polymerization, the specimen was immediately removed from the mold and four Raman spectra were acquired from the middle area of the top and bottom surface using micro-Raman spectroscopy (μRaman; Senterra, Bruker, Ettlingen, Germany). The surface was excited with a near-infrared (785 nm) laser of 50 mW and analyzed through a 100× objective and 50-μm pin-hole aperture. The collected spectra ranged from 50 to 3,500 cm⁻¹ with a resolution of 9–15 cm⁻¹. The integration time of each spectrum was set to 20 s with 2 co-additions. The CCD detector to obtain the

Table 2

Characteristics of the dental monomers and their derivatives investigated.

Molecule	Name	Molecular formula	Molecular weight	CAS-number	Supplier
BisGMA	Bisphenol A-glycidyl methacrylate	C ₂₉ H ₃₆ O ₈	512.6	1565-94-2	Sigma-Aldrich ^a
BisEMA(6)	Bisphenol A ethoxylateddimethacrylate	C ₃₅ H ₄₈ O ₁₀	628.7	41637-38-1	ESSTECH Inc ^b
BisPMA	2,2-Bis-(4-(3 methacryloxypropoxy) phenyl) propane	C ₂₉ H ₃₆ O ₆	480.59	27689-12-9	3M ESPE ^c
UDMA	Urethane dimethacrylate	C ₂₃ H ₃₈ N ₂ O ₈	470.56	72869-86-4	Sigma-Aldrich
TEGDMA	Triethylene glycol dimethacrylate	C ₁₄ H ₂₂ O ₆	286.32	109-16-0	Sigma-Aldrich
BPA	Bisphenol A	C ₁₅ H ₁₆ O ₂	228.29	80-05-7	Sigma-Aldrich
d16-BPA ^d	Deuterated (d16) bisphenol A	C ₁₅ D ₁₆ O ₂	244.38	96210-87-6	Sigma-Aldrich
d4-diethyl phthalate ^d	Deuterated (d4) diethyl phthalate	C ₆ D ₄ C ₆ H ₁₀ O ₄	226.26	93952-12-6	Cambridge Isotope Laboratories ^e

^a St Louis, MO, USA.^b Fremont, CA, USA.^c Seefeld, Germany.^d Added to solvent as internal standard for LC/MS-MS analysis.^e Andover, MA, USA.

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