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Analysis of long-term monomer elution from bulk-fill and conventional resin-composites using high performance liquid chromatography

Ruwaida Z. Alshali^{a,b}, Nesreen A. Salim^c, Rehana Sung^d,
Julian D. Satterthwaite^a, Nick Silikas^{a,*}

^a School of Dentistry, The University of Manchester, Manchester, UK

^b Department of Oral and Maxillofacial Rehabilitation, King Abdulaziz University, Jeddah, Saudi Arabia

^c Prosthodontic Department, University of Jordan, Amman, Jordan

^d Manchester Institute of Biotechnology, The University of Manchester, Manchester, UK

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ABSTRACT

Objective. The aim of this study was to assess monomer elution from bulk-fill and conventional resin-composites stored in different media using high performance liquid chromatography (HPLC) for up to 3 months.

Methods. Six bulk-fill (SureFil SDR, Venus Bulk Fill, X-tra base, Filtek Bulk Fill flowable, Sonic Fill, and Tetric EvoCeram Bulk Fill) and eight conventional resin-composites (Grandioso Flow, Venus Diamond Flow, X-Flow, Filtek Supreme XTE, Grandioso, Venus Diamond, TPH Spectrum, and Filtek Z250) were tested. Cylindrical samples ($n = 5$) were immersed in water, 70% ethanol/water solution (70% E/W), and artificial saliva and stored at 37 °C for 24 h, 1 month, and 3 months. The storage solutions were analysed with HPLC. Data were analysed with repeated measures ANOVA, one-way ANOVA, and Tukey post hoc test at $\alpha = 0.05$.

Results. Monomers detected in water and artificial saliva were TEGDMA, DEGDMA, UDMA, and TCD-DI-HEA. No eluted monomers were detected from X-tra base and Sonic fill in these media. All monomers showed a variable extent of elution into 70% E/W with significantly higher amounts than those detected in water and artificial saliva. Significantly higher elution was detected from UDMA-BisEMA based composites compared to BisGMA and BisGMA-BisEMA based systems in 70% E/W. The rate of elution into different media varied between different monomers and was highly dependent on the molecular weight of the eluted compounds.

Significance. Elution from bulk-fill resin-composites is comparable to that of conventional materials despite their increased increment thickness. Monomer elution is highly dependent on the hydrophobicity of the base monomers and the final network characteristics of the resin-matrix.

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* Corresponding author at: School of Dentistry, The University of Manchester, Manchester M13 9PL, UK. Tel.: +44 1612756747.
E-mail address: nick.silikas@manchester.ac.uk (N. Silikas).

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1. Introduction

Dental resin-composites are considered stable restorative materials, however they are susceptible to degradation and leaching out of fractions of their components [1]. Monomer conversion in light-cured resin-composite systems is never complete and it varies between 40 and 75%: double bonds remain as pendant groups and free monomer molecules trapped in the cross-linked polymer network [2]. Free monomers account for 10% of unconverted double bonds and may elute from resin-composites [2,3]. The amount of eluting species ranges between 0.5 and 2% weight in water, 2–6% weight in 70% ethanol, and 10% in methanol [1,3–5]. More than 30 chemical substances have been found to be released from dental resin-composites into different storage media [6] including residual monomers, oligomers, initiators, catalysts, polymerization stabilizers, passive hydrolysis and biodegradation products [7,8], polymerization products [9], impurities, and metal ions [10].

Since monomers constitute the main part of a resin matrix (20–40 wt%), they may represent the largest risk for biotoxic effects and weakening of the mechanical properties upon elution [11]. Although some studies have shown that the extent of monomer elution is correlated to the degree of double bond conversion [12,13], the degree of conversion measured by FTIR does not necessarily correlate with the amount of free residual monomer since the detected double bonds may remain as pendant groups which are bonded to the polymer structure and are not free to leach out [1]. In addition to their effect on mechanical properties such as decreased wear resistance, hardness, and increased tendency to discoloration, eluted monomers can contribute to a variety of local or systemic health effects. They can be released either into the oral cavity or diffuse into the pulp through dentinal tubules causing local reactions including pulpal irritation [14,15], allergenic, cytotoxic, and genotoxic effects [16–20]. Eluted TEGDMA has been shown to promote the growth of cariogenic bacteria [21]. Some eluted moieties have been linked to systemic effects: Bisphenol A (BPA), a hydrolytic degradation product or a contaminant eluted from aromatic based systems has been confirmed to have parahormonal activity and it can imitate hormones from the estrogen group and thus may contribute to female infertility [22,23].

A residual monomer or a compound can leach out of a polymeric network only when two conditions exist: diffusion and swelling [13,24]. Diffusion occurs when the solubility parameter of the storage solution matches that of the polymer structure which is mainly affected by the degree of hydrophobicity of the polymer structure. Aqueous solvents are attracted by hydrophilic networks while organic solvents diffuse more easily into hydrophobic structures. Diffusion into a polymeric network results in swelling and opening up of existing pores. The degree of swelling depends on the rigidity and cross-link density of the polymer network and the diffusion of residual monomer out of the polymer depends on its molecular weight and flexibility. Small low molecular weight monomers such as TEGDMA diffuse easily and at a higher rate compared to bulky molecules with a rigid structure such as BisGMA and BisEMA monomers [25].

Recently, bulk-fill resin-composites have been introduced into the market; these show adequate degree of conversion and depth of cure when placed in a single increment of 4–5 mm thickness compared to a maximum of 2 mm increment for conventional materials [26,27]. The enhanced depth of cure of bulk-fill resin-composites has been related to their increased translucency [28] in addition to the incorporation of polymerization boosters and altered resin composition [29]. Although bulk-fill resin-composites have been assessed extensively and compared to conventional resin-composites in terms of degree of cure and physico-mechanical performance [30–34], the literature is lacking data regarding monomer elution from these materials in different storage solutions. In view of the limited research in this area, the aim of this study was to assess monomer elution from light-cured bulk-fill and conventional resin-composites stored in different media over a three-month period using a high performance liquid chromatography (HPLC) technique. The null hypothesis was that there would be no difference in the extent of monomer elution between bulk-fill and conventional resin-composites over time in different storage media.

2. Material and methods

2.1. Solvents and reagents

All solvents in this study were HPLC grade. Water, ethanol, acetonitrile, caffeine (CF), components of artificial saliva, Bisphenol A ethoxylate dimethacrylate (BisEMA), Diethylene glycol dimethacrylate (DEGDMA), and Ethoxylated bisphenol A diacrylate (EBPADA) were from Sigma-Aldrich, UK. Bisphenol A glycidyle dimethacrylate (BisGMA), triethylene glycol dimethacrylate (TEGDMA), and urethane dimethacrylate (UDMA) were supplied by Röhm GmbH, Germany.

2.2. Sample preparation

Fourteen commercial resin-composite materials including six bulk-fill materials and eight conventional resin-composite materials were tested. A list of the resin-composites studied is given in Table 1. Cylindrical samples were made using a 4 mm diameter × 4 mm height polytetrafluoroethylene (PTFE) mould. The mould was placed against a cellulose acetate matrix strip and a glass slab on a non-reflective background surface. Samples of bulk-fill materials were applied in a single bulk increment into the mould while conventional materials were applied in two increments, each of 2 mm thickness. The mould was slightly overfilled with material and any excess was then extruded by applying another matrix strip and a glass slab with firm pressure. Each sample was then cured from the top surface for 20 s using a LED light-curing unit (Elipar™, 3M ESPE, USA) under standard curing mode. The light-curing unit had an output irradiance of 1200 mW/cm² and wavelength range 430–480 nm. A calibrated radiometer system (MARC Blue-light Analytics Inc, Halifax, NS, Canada) was used to verify the irradiance at each use. Immediately after cure, each sample was gently pushed out from the mould and the excess flash of the material was removed using a sharp blade.

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