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Qualitative and quantitative characterization of monomers of uncured bulk-fill and conventional resin-composites using liquid chromatography/mass spectrometry

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ABSTRACT

Objectives. The aim of this study was to assess the resin matrix monomer composition of selected bulk-fill and conventional resin-composite materials using reverse phase liquid chromatography coupled with electron spray ionization mass spectrometry.

Material and 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. For assessment of resin composition and relative monomer amounts, uncured resin-composites were analysed with reverse phase liquid chromatography/electron spray ionization mass spectrometry. $\log P$ values (a measure of hydrophobicity) of detected compounds were calculated and their correlation to reverse phase liquid chromatography retention time was explored. Data were analysed with one-way ANOVA, Tukey post hoc test, Pearson correlation and regression analyses at $\alpha = 0.05$.

Results. The main monomers detected were BisGMA, UDMA, TEGDMA, and BisEMA. Monomers were detected at variable combinations in different materials with significantly different relative amounts. Other monomers were detected including HDDMA, DEGDMA, TCD-DI-HEA, and SDR-UDMA in Grandioso flow, X-flow, Venus Diamond, and SureFil SDR respectively. A positive correlation between $\log P$ and reverse phase liquid chromatography retention time was detected ($r^2 = 0.62$, $p = 0.004$).

Conclusions. Resin composition of bulk-fill resin-composites is comparable to that of conventional materials with the exception of SureFil SDR. The relative hydrophobicity of dental monomers can be determined by their reverse phase liquid chromatography retention time.

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1. Introduction

Resin-composites are widely used dental materials since they restore both function and esthetics of dental tissues [1,2]. The main components of a resin-composite are the inorganic fillers, and the organic resin matrix. The resin matrix constitutes about 20–40 wt% of a resin-composite material and is mainly composed of monomeric compounds including base monomers, co-monomers and additives [3]. Bowen in the early 1960s introduced the dimethacrylate monomer Bisphenol A Glycidyl Methacrylate (BisGMA) as the first monomer suitable to be incorporated into a resin-composite formulation intended for use as a direct dental restorative material [4]. Dental resin-composites have evolved significantly since they were first introduced to improve their strength, wear resistance, and polymerization shrinkage properties; although most of the changes have involved the inorganic filler fraction [5]. The resin fraction; comprised mainly of the base monomer BisGMA; has largely remained unchanged. BisGMA is a highly viscous monomer due to its hydroxyl groups which increase its polarity and result in strong intermolecular attraction [6]. It is usually diluted with a low molecular weight linear monomer triethylene glycol dimethacrylate (TEGDMA). Dilution of BisGMA with TEGDMA reduces its viscosity, improves its handling properties, degree of conversion [7–9], and allows more filler fraction to be loaded into the resin. However, BisGMA dilution with TEGDMA has been shown to be associated with increased polymerization shrinkage and water sorption [10,11].

Urethane dimethacrylate monomer (UDMA) is a low viscosity base monomer that has molecular weight close to that of BisGMA, however, the absence of the phenol rings in UDMA results in a more flexible structure with higher toughness in comparison to BisGMA [12,13]. Ethoxylated bisphenol A dimethacrylate (BisEMA) is a BisGMA analogue with low viscosity due to the absence of hydroxyl groups [6]. BisEMA is available in different molar weights depending on the ethylene oxide chain length between the aromatic core and the functional methacrylate groups and increasing the degree of ethoxylation of BisEMA further reduces its viscosity and increases conversion, but also increases water sorption and decreases flexural strength [6,14]. Variable combinations and proportions of monomers are used in current resin-composites resulting in different copolymer systems. The long-term hygroscopic and hydrolytic stability of these materials has been shown to be dictated by the hydrophobicity of their constituent monomers, and by the final network characteristics; specifically, the degree of conversion, cross-link density and structural heterogeneity of their copolymer structure [15]. To have a better understanding and assessment of their effect on the clinical and laboratory performance, adequate qualitative and quantitative data about a resin-composite constituent monomers and co-monomers is mandatory. Unfortunately, due to commercial protectionism, these data are usually either missing or not completely disclosed by the manufacturers [3].

The synthesis of new dental monomers is mainly driven by the aim of reducing polymerization shrinkage, increasing the biocompatibility of the materials, and maximizing polymerization potential. Recently, a new class of materials described

as ‘bulk-fill’ resin-composites has been introduced. Bulk-fill resin-composites are mainly characterized by their enhanced depth of cure allowing thick increments (4–5 mm) to be placed in a single step instead of the conventional layering technique [16,17]. One such material (SureFil® SDR®) is based on a modified UDMA stress-decreasing resin system with an incorporated polymerization modulator photoactive group [18].

Determination and quantification of different dental resin components and eluted species has been carried out using the sensitive techniques of gas chromatography (GC) and high performance liquid chromatography (HPLC) [19]. Both HPLC and GC can be coupled to a mass spectrometer (MS) as a detector to increase the sensitivity and selectivity of the technique. This also allows identification of unknown substances and degradation products from both uncured resins and extracts from cured materials [20]. Less commonly used techniques include fluorescence spectrophotometry [21] and micellar electrokinetic chromatography [22].

The aim of this study was to assess the resin-matrix monomer composition of selected bulk-fill and conventional resin-composite materials, using liquid chromatography/mass spectrometry to identify the constituent monomers of the uncured resin of these materials and their relative proportions. The first null hypothesis was that there would be no difference in the type and relative proportions of monomers that constitute the uncured resin of bulk-fill and conventional resin-composite materials. The hydrophobicity of identified monomers and compounds was also assessed by generating their predicted octanol–water partition coefficient ($\log P$), and the correlation between predicted $\log P$ values of monomers and their reverse phase liquid chromatography retention time was explored. Hence, the second null hypothesis was that there would be no correlation between predicted $\log P$ values and reverse phase liquid chromatography retention time of the identified compounds.

2. Materials and methods

2.1. Solvents and reagents

All solvents in this study were HPLC grade. Water, methanol, acetonitrile, caffeine, BisEMA, DEGDMA, EBPADA, and MMA were from Sigma–Aldrich, UK. BisGMA, TEGDMA, UDMA, HEMA were supplied by Röhm GmbH, Germany (Table 2).

2.2. Characterization of uncured resin

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. Three samples of each uncured resin-composite material were prepared. For each sample 0.1 g of uncured resin-composite was dissolved in 1.5 ml methanol (containing 0.1 mg/ml caffeine as an internal standard). The samples were shaken for 10 min until the composite was completely dissolved in the methanol solution. The samples were then centrifuged at 15,000 *g* for one minute to separate the filler particles from the solution (IEC MicroCL 17 centrifuge, Thermo Electron Corporation, USA). The supernatant

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