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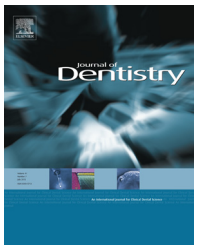

Influence of various irradiation processes on the mechanical properties and polymerisation kinetics of bulk-fill resin based composites

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ARTICLE INFO

Article history:
Received 18 January 2013
Received in revised form
13 May 2013
Accepted 14 May 2013

Keywords:
Bulk-fill resin based composites
Irradiance
Hardness
Modulus of elasticity
Polymerisation kinetic

ABSTRACT

Objectives: To assess the effect of irradiation time and distance of the light tip on the micro-mechanical properties and polymerisation kinetics of two bulk-fill resin-based composites at simulated clinically relevant filling depth.

Methods: Micro-mechanical properties (Vickers hardness (HV), depth of cure (DOC) and indentation modulus (*E*)) and polymerisation kinetics (real-time increase of degree of cure (DC)) of two bulk-fill resin-based composites (Tetric EvoCeram® Bulk Fill, Ivoclar Vivadent and x-tra base, Voco) were assessed at varying depth (0.1–6 mm in 100 μm steps for *E* and HV and 0.1, 2, 4 and 6 mm for DC), irradiation time (10, 20 or 40 s, Elipar Freelight2) and distances from the light tip (0 and 7 mm). Curing unit's irradiance was monitored in 1 mm steps at distances up to 10 mm away from the light tip on a laboratory-grade spectrometer.

Results: Multivariate analysis ($\alpha = 0.05$), Student's *t*-test and Pearson correlation analysis were considered. The influence of *material* on the measured mechanical properties was significant ($\eta^2 = 0.080$ for *E* and 0.256 for HV), while the parameters *irradiation time*, *distance from the light tip* and *depth* emphasise a stronger influence on Tetric EvoCeram® Bulk Fill. The polymerisation kinetics could be described by an exponential sum function, distinguishing between the gel and the glass phase. The above mentioned parameters strongly influenced the start of polymerisation (gel phase), and were of less importance for the glass phase.

Conclusions: Both materials enable at least 4 mm thick increments to be cured in one step under clinically relevant curing conditions.

Clinical significance: The susceptibility to variation in irradiance was material dependent, thus properties measured under clinically simulated curing conditions might vary to a different extent from those measured under ideal curing conditions.

1. Introduction

In an attempt to speed up the restoration process, a new resin-based composite (RBC) material class, the bulk-fill RBCs, was recently introduced on the market, enabling up to 4 or 5 mm thick increments to be cured in one step, thus skipping the time-consuming layering process. The mechanical stability in stress bearing areas of fillings restored with bulk-fill RBCs is still an open question, since long-term clinical studies are not

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0300-5712/$ – see front matter
http://dx.doi.org/10.1016/j.jdent.2013.05.008

available so far. *In vitro* studies revealed for bulk-fill RBCs, as a material class, similar flexural strength values as the class of nano- and micro-hybrid RBCs, and significantly higher values when compared to flowable RBCs. The modulus of elasticity, the indentation modulus and the hardness classify this materials as between the hybrid RBCs and the flowable RBCs, while in terms of creep, bulk-fill and flowable RBCs perform similar, both showing a significantly lower creep resistance when compared to the nano- and micro-hybrid RBCs.[1] Other studies found, however, that bulk-fill RBCs exhibited a creep deformation within the range of regular RBCs.[2]

A main concern when curing large increments remains however potentially increased polymerisation shrinkage stress at the tooth-material interface.[3,4] Few studies are available so far to allow a critical assessment of bulk-fill materials placed in deep cavities. Compared to regular flowable and non-flowable nano- and micro-hybrid methacrylate-based RBCs and a silorane-based micro-hybrid RBC, a bulk-fill material in its experimental version (SDR®, Dentsply) revealed the lowest shrinkage stress and shrinkage-rate values in 2 mm (thick) × 4 mm (width) × 4 mm (length) cavities.[5,6] Corroborative to this study, shown bulk-fill flowable RBCs (SDR®, Dentsply; x-tra base, VOCO) significantly reduced cuspal deflection in standardised Class II cavities in comparison with a conventional RBC (GrandioSO, VOCO) restored in an oblique incremental filling technique.[7] Moreover, flowable bulk-fill RBCs, like SureFil SDR, are considered to be adequate for both, post cementation and core build-up.[8]

The alleged changes in the rheology of bulk-fill RBCs compared to regular RBCs that are supposed to allow a better adaption to the cavity walls remained unconfirmed by in vitro microleakage[7] and marginal integrity studies.[9] The material's reliability however, a characteristic associated with lower surface defects that are able to initiate crack propagation, proved to be very high in low viscosity bulk-fill RBCs (high values for the Weibull modulus), but moderate, and hence comparable to regular nano- and micro-hybrid RBCs in high viscosity RBCs.[1] This behaviour proved, indirectly a better adaptability to the cavity (or mould) surface, in low viscosity bulk-fill RBCs. The classification in low and high viscosity bulk-fill RBCs is unambiguously reflected in the mechanical properties[1] and determines the application procedure of the materials. Restorations made with low viscosity bulk-fill RBCs (SureFil®SDR®, Dentsply; Venus® Bulk Fill, Heraeus Kulzer; x-tra base, VOCO; Filtek™ Bulk Fill, 3M ESPE) must be finished by adding a capping layer made of regular RBCs, while high viscosity bulk-fill RBCs (SonicFill™, Kerr; Tetric EvoCeram® Bulk Fill, Ivoclar Vivadent; X-tra Fil, VOCO) may be placed without capping.

In vitro studies confirmed that bulk-fill RBCs could be cured in larger increments, as the degree of cure (DC) and the micro-mechanical properties were maintained within 4 mm layers at an irradiation time of up to 20 s (SDR®, Dentsply; Venus Bulk Fill®, Heraeus Kulzer).[10] A reason for an enhanced depth of cure is considered to be an increased translucency, due to decreased filler load and increased filler size,[1] since the chemical composition and the initiator systems (except for Tetric EvoCeram® Bulk Fill, Ivoclar Vivadent) in bulk-fill RBCs do not consistently differ from regular RBCs.[1]

An important aspect, often ignored in depth-of-cure-studies, where the light tip of the curing unit is placed directly against the material, is the consistent decrease of irradiance in correlation with the distance from the light tip, which, in a clinical situation, is frequently at least 7 mm.[11,12]

This study therefore aims to evaluate the effect of polymerisation time and distance from the light tip on two bulk-fill RBCs – Tetric EvoCeram® Bulk Fill and x-tra base – by assessing their micro-mechanical properties and polymerisation kinetics (variation of the degree of cure), at simulated clinical relevant filling depths.

The tested null hypotheses were that: (a) there would be no significant differences between the two materials in view of micro-mechanical properties (Vickers hardness (HV), depth of cure (DOC), indentation modulus (*E*)), degree of conversion (DC) or polymerisation kinetics, at any measured depth, irradiation time or distances away from the light tip and (b) within one material, the above mentioned parameters would not influence the measured properties.

2. Materials and methods

Two bulk-fill RBCs (Table 1) were investigated by assessing DC and micro-mechanical properties (HV, DOC, *E*) as function of depth (0.1, 2, 4 and 6 mm for DC measurements and 0.1–6 mm in 100 μm steps for *E* and HV), polymerisation time (10, 20 or 40 s) and distances away from the light tip (0 and 7 mm).

2.1. *Degree of conversion (DC)*

DC was measured in a real-time profile (5 min, with 2 spectra/s) with an FTIR-Spectrometer with an attenuated total reflectance (ATR) accessory (Nexus, Thermo Nicolet, Madison, USA). Four different sample geometries were considered. Thin films (100 μm) as well as 2, 4 and 6 mm high moulds (3 mm diameter) were filled in bulk. Samples were cured by applying the curing unit (Elipar Freelight2, 3M ESPE, Fig. 1) directly on the top (0 mm) and in 7 mm distance from the particular mould, respectively, the film surface covered by a transparent matrix strip. For each material (Tetric EvoCeram® Bulk Fill, x-tra base), irradiation time (10, 20 and 40 s), thickness (0.1, 2, 4 and 6 mm) and distance from the light tip (0 and 7 mm), summarising 48 groups, six samples were measured ($n = 6$). The non-polymerised composite paste was applied directly on the diamond ATR crystal in the mould as described above. DC was measured on the bottom of the samples and calculated, in relation to the uncured material, by assessing the variation in peak height ratio of the absorbance intensities of methacrylate carbon–carbon (C–C) double bond (peak at 1634 cm^{-1}) and that of an internal standard (aromatic C–C double bond, peak at 1608 cm^{-1}) during polymerisation.

$$DC_{peak}\,\% = \left[1 - \frac{(1634\ cm^{-1}/1608\ cm^{-1})_{\text{peak height after curing}}}{(1634\ cm^{-1}/1608\ cm^{-1})_{\text{peak height before curing}}}\right] \times 100$$

In each sample, the increase in DC (=decrease of the C–C double bonds) was described by the superposition of two exponential functions, the first: $a \times (1 - e^{-bx})$ being attributed